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NAVAL AIR WARFARE CENTER AIRCRAFT DIVISION
PATUXENT RIVER, MARYLAND



TECHNICAL REPORT

REPORT NO: NAWCADPAX/TR-2011/162

HIGH NITROGEN STAINLESS STEEL

by

**E. U. Lee
R. Taylor**

19 July 2011

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RELEASED BY:

A handwritten signature in black ink, appearing to read 'C Matzdorf', is positioned above the printed name and title.

19 JUL 2011

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| 14. ABSTRACT A study was conducted to clarify the mechanical properties and stress corrosion cracking (SCC) resistance of high nitrogen stainless steel (HNSS) plates, produced by powder metallurgy – rapid solidification process. It was found that the HNSS was highly workable and the yield strength, ultimate tensile strength, hardness, elongation, fracture toughness, and fatigue behavior were influenced by high nitrogen concentration and cold-work. Furthermore, the HNSS, annealed or cold-worked, was immune to SCC. | | | | | |
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SUMMARY

An investigation was carried out to characterize the influence of high nitrogen (N) concentration and cold-work on the mechanical properties, fatigue resistance, and stress corrosion cracking (SCC) susceptibility of high nitrogen stainless steel (HNSS). A set of HNSS plates, produced by means of powder metallurgy – rapid solidification process, was employed as the specimen material. The set consisted of annealed and 28, 35, 45, and 50% cold-worked plates. The specimens from these plates were subjected to metallurgical examination and tension, stress-life fatigue, fatigue crack growth, and SCC tests. The results indicate: (a) with increasing amount of cold-work, the yield strength, ultimate tensile strength, and hardness increase, the elongation and fracture toughness decrease, and the fatigue resistance becomes greater in air and 3.5% NaCl solution; (b) the HNSS is immune to SCC but susceptible to corrosion fatigue in 3.5% NaCl solution; and (c) N contributes to the enhancement of mechanical properties, cold-workability, and resistance to fatigue and SCC.

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INTRODUCTION

Nitrogen (N) in iron and steels has been studied for nearly 100 years, beginning in 1912 at Carnegie Institute (reference 1). In the 1920s and 1930s, the study was carried out primarily in Germany (reference 2). Subsequently, Armco developed the Nitronic series of N containing steels, designed for a specific purpose, such as high strength, wear resistance, or improved corrosion resistance (reference 3). The extensive studies in the following years accumulated the understanding of the beneficial effects of N on iron and steels (references 4-8). Among the most significant were studies on mechanical properties (references 9 and 10), N solubility (references 1, 11, 12, and 13), thermodynamics (references 2 and 11), corrosion resistance (reference 14), and precipitation of intermetallics (references 11 and 15). However, several factors limited the potential use of N containing iron and steels. Those factors include the following:

- the brittle, stable nitrides and intermetallics, formed during the slow cooling in the process of ingot casting or hot isostatic press (HIP) consolidation of atomized powders
- the other casting defects, such as macrosegregation, which make the iron and steels unworkable by conventional wrought process
- the difficulty in producing predictable N concentrations during conventional and pressurized casting

National Institute of Standards and Technology and the other organizations (references 9 and 10) demonstrated that these problems could be overcome, employing the powder metallurgy – rapid solidification process (PM-RSP) technique with N gas atomization and HIP consolidation (reference 16). This technique introduced additional benefits, such as increased N concentration, enhanced microstructural refinement, and chemical homogeneity. Furthermore, the brittle Cr_2N and sigma phase intermetallics were reduced or eliminated and the porosity was less than found in castings (references 16 and 17).

Alloying with N in solid solution confers favorable properties on steels: it stabilizes the face-centered cubic lattice and enhances the resistance to pitting, intergranular, and stress corrosion. In addition, the N alloying enhances yield strength (YS), ultimate tensile strength (UTS), and hardness and improves creep and fatigue resistance. These effects, together with the N-induced grain refinement and high workability, play an essential role in the strengthening process of austenitic stainless steels. It has also been reported that a cold-work makes further improvement of mechanical properties possible.

A study was conducted to clarify the influence of N and cold-work on the mechanical properties and stress corrosion cracking (SCC) resistance of high nitrogen stainless steel (HNSS). The results are compiled into this report.

EXPERIMENTAL PROCEDURE

MATERIAL

A set of HNSS plates, produced by means of the PM-RSP process (reference 16), was supplied by Carpenter Technology Corp. The set consisted of annealed and 28, 35, 45, and 50% cold-worked plates. The annealing was done by heating at 2000°F (1093°C) for 1 hr and subsequent water quenching. The cold-working was done by rolling and reducing the thickness at room temperature. The chemical composition of the HNSS is shown in Table B-1, and the microstructures of the annealed and 35 and 50% cold-worked plates are shown in Figure A-1.

SPECIMENS

The HNSS plates were machined to the following specimens:

- round tension test specimen of gage section diameter 0.16 in. in L orientation for tension test (ASTM E8-99)
- hourglass specimen of minimum diameter 0.125 in. in L-orientation for stress-life fatigue test (ASTM E 466-99)
- compact tension specimen, 1.5 in. wide and 0.125 in. thick, in L-T crack plane orientation for fatigue crack growth (FCG) test (ASTM E 647-95a)
- square bar specimen of 0.4x0.4x2.8 in. in L-orientation with a Charpy notch at the mid-length for SCC test under four-point bending (ASTM F 1624-95)

TESTS

Tension Test: A closed-loop servo-hydraulic mechanical test machine, Interlaken, of 20 kip capacity was utilized for the tension test. The test was conducted with the round tension test specimen in air, following the ASTM E 8/E-8M – 08, Standard Test Methods for Tension Testing of Metallic Materials. The tensile loading rate was 0.003 in./min.

Stress-Life Fatigue Test: This test was also carried out in the Interlaken of 20 kip capacity, employing the hourglass specimen. The fatigue cycling was done under stress control at stress ratio 0.1 and frequency 10 Hz in air and aqueous 3.5% NaCl solution of pH 7.3. This test followed the ASTM E 466 – 07, Standard Practice for Conducting Force Controlled Constant Amplitude Axial Fatigue Tests of Metallic Materials.

Fatigue Crack Growth Test: A horizontal closed-loop servo-hydraulic mechanical test machine of 10 kip capacity was used for the fatigue crack growth test. The test was performed under stress control in tension-tension cycling of frequency 10 Hz and stress ratio 0.1 in air and aqueous 3.5% NaCl solution of pH 7.3 at room temperature. The fatigue loading procedure was K-decreasing or load shedding with K-gradient parameter $C = -1 \text{ in.}^{-1}$ in the near threshold FCG regime and K-increasing in the Paris and rapid unstable crack growth regimes. Using compliance

technique, the fatigue crack length was continuously monitored with a laboratory computer system, interfaced with the test machine. This test followed the ASTM E 647 – 08, Standard Test Method for Measurement of Fatigue Crack Growth Rates.

Stress Corrosion Cracking Test: Since the cantilever bend or the double-cantilever beam SCC test takes a long time, an accelerated SCC test was conducted in a RSL 1000 SI-Multi-Mode Test System (reference 18). This system included a bending frame, a tensile loading frame, an electrolyte reservoir, a pump for electrolyte circulation, a saturated calomel electrode, a platinum counterelectrode, a PC, and a printer. The precracked specimen was step-loaded until the load dropped in four-point bending under constant displacement control, while held at a given potential in aqueous 1% NaCl solution of pH 2 or 3.5% NaCl solution of pH 7.3. From the load drop at the open circuit potential, the threshold stress intensity for stress corrosion crack growth, K_{ISCC} , was determined. This test followed the ASTM F 1624 – 95, Standard Test Method for Measurement of Hydrogen Embrittlement in Steel by the Increment Loading Technique.

Fractography: After the stress-life fatigue tests, the fracture surface morphology was examined with a scanning electron microscope, JEOL JSM-6460LV, operated at an accelerating voltage of 20 kV.

EXPERIMENTAL RESULTS

MECHANICAL PROPERTIES

The determined mechanical properties of the annealed and cold-worked HNSS are listed in Table B-2. The variation of mechanical properties with cold-work is illustrated in Figure A-2. With increasing amount of cold-work, the YS, UTS, and hardness increased, but the elongation and fracture toughness decreased, indicating work-hardening.

FATIGUE BEHAVIOR

STRESS-LIFE FATIGUE

Stress-life fatigue curves are shown in Figure A-3 for the annealed and 28% and 50% cold-worked specimens, fatigue-tested in air and 3.5% NaCl solution of pH 7.3. In both environments, the fatigue resistance is greatest for the 50% cold-worked specimen and least for the annealed one. The fatigue lives in air and 3.5% NaCl solutions are compared in Figure A-4. For the annealed and 28% and 50% cold-worked specimens, the fatigue life is shorter in 3.5% NaCl solution than in air under a given maximum applied stress.

FATIGUE CRACK GROWTH

Figure A-5 shows the fatigue crack growth rate, da/dN , versus the stress intensity range, ΔK , curves for the annealed and 28% and 50% cold-worked specimens in air and 3.5% NaCl solution. In both of the environments, the three curves nearly overlap each other. However, the cold-work appears to accelerate slightly the fatigue crack growth, reduce the threshold stress intensity range for fatigue crack growth, ΔK_{th} , and shorten the fatigue crack growth life,

compared to the annealing. This trend is more obvious for a greater cold-work. Since the stress-life curves (Figure A-3) show that the total fatigue life is longer for a greater cold-work under a given maximum applied stress, the fatigue crack initiation life must be longer for the cold-worked specimen. In other words, cold-work extends the fatigue crack initiation and total fatigue lives, but shortens the fatigue crack growth life of HNSS.

Figure A-6 compares the fatigue crack growth rates, da/dN , in air and 3.5% NaCl solution for the annealed and 28% and 50% cold-worked specimens. The da/dN is greater and the ΔK_{th} is less in 3.5% NaCl solution than in air.

RESISTANCE TO STRESS CORROSION CRACKING

The threshold stress intensity for stress corrosion cracking, K_{ISCC} , of HNSS, annealed, and cold-worked, was so large that it was not measurable by means of the Rising Step Load method in a RSL 1000 SI-Multi-Mode Test System. In other words, the K_{ISCC} of HNSS is beyond the measurable limit, and HNSS is not susceptible to SCC in 1% NaCl solution of pH 2 or 3.5% NaCl solution of pH 7.3.

FRACTOGRAPHIC FEATURES

The SEM fractographs for the annealed and 50% cold-worked specimens, subjected to the stress-life fatigue tests in air and 3.5% NaCl solution, are shown in Figures A-7 and A-8, respectively. The main fractographic features are observed to be patches of fatigue-striations and some scattered dimples without noticeable difference in those fractographs. This observation indicates that the specimen annealing and cold-working, and the test environments, air and 3.5% NaCl solution, do not induce a noticeable change in fractographic features, whereas they influence the fatigue life and strength noticeably.

DISCUSSION

ROLE OF NITROGEN

When the yield strengths of stainless steels with different N concentrations are compared, the dominant contribution to the YS is found to be the interstitial N concentration. As long as N is interstitial, the YS is reported to increase with the square root of the N concentration, or the flow stress is roughly a linear function of N concentration (reference 19).

N forms an interstitial solid solution in austenite, brings about solid solution strengthening, and favors the planar dislocation slip (reference 20). The strengthening is attributable to the interaction of moving dislocations with the stress field around N atoms in the interstices of the fcc crystal lattice. N also induces grain refinement, which results in polycrystalline strengthening, following the Hall-Patch relation $\sigma_y = \sigma_o + kd^{-1/2}$, where σ_y is the YS and d the grain size. The planarity of the dislocation slip is the reason for the grain boundaries becoming more effective obstacles to the slip transfer from one grain to the other.

A high N concentration also raises the work-hardening coefficient, makes cold-working more efficient, and enhances the mechanical properties. Furthermore, a high N content improves the resistance of steels to corrosion and stress corrosion cracking. Within certain limits, 1% N is as effective as 30% Cr in preventing pitting corrosion by raising the critical temperature where pitting corrosion starts (reference 20). The reported enhancement of mechanical properties, arising from high N concentration and cold-work, is validated by the results of this study, as shown in Tables B-1 and B-2 and Figure A-2.

N improves the fatigue life of austenitic steels; most studies attributed this improvement to increased resistance to strain localization from increased planar slip. Resistance to strain localization reduces the formation of persistent slip bands and increases slip reversibility (reference 21). The result of this study, Figure A-3, indicates that a greater cold-work extends the fatigue life of HNSS, containing 0.85% N, more.

MICROSTRUCTURE

The noticeable microstructural features of annealed and 35% and 50% cold-worked HNSS plates are as follows:

- austenite matrix
- annealing twins in the annealed plate and deformation twins in the cold-worked ones
- smaller grain size in cold-worked ones
- small amount of scattered precipitated particles, possibly chromium nitride Cr_2N and/or sigma phase
- absence of deformation-induced martensite

Reportedly, N assists the twinning of austenite on $\{111\}$ planes under deformation. Therefore, an increase in N concentration strengthens the austenitic steels further with the greater planar slip and higher density of deformation twin (references 22 and 23).

Cr_2N and sigma phase precipitation reduces the ductility and detract the corrosion resistance. It is therefore necessary to dissolve those precipitates into solution by a proper heat treatment.

CORROSION RESISTANCE

N usually reduces the deleterious sensitization and increases the corrosion resistance (references 24 and 25). Besides, N actively assists the formation of a preliminary film and, apparently, subsequent passive films containing more Cr and Mo (reference 26). N decreases the rate of pitting corrosion, resulting from localized breakdown of such a passive film (reference 21). Truman (reference 27) reviewed various studies relating N content to stress corrosion and corrosion fatigue and concluded that increase in N level reduces the effects of stress-enhanced corrosion. However, if the N content exceeds its solubility limits, Cr_2N will

precipitate and deplete the matrix of Cr, thus reducing passivity. Grabke (reference 14) reported that Cr-Mn-N steels exhibit not only a good combination of strength and toughness, but also increasing resistance to SCC with increasing N content.

COMPARISON WITH SOME STAINLESS STEELS

The mechanical properties and K_{ISCC} values of the HNSS are compared with those of the other stainless steels, Custom 465, 13-8Mo, and MLX17, in Table B-3. The UTS and YS of the HNSS, annealed or cold-worked, are lower than those of Custom 465, 60% cold-worked and aged at different temperatures. However, some of them are similar to those of 13-Mo (H1000) and MLX17, depending on the amount of cold-work.

The stress-life fatigue and fatigue crack growth behaviors of the HNSS are compared with those of the other stainless steels, Custom 465, 13-8Mo, and MLX17, in Figures A-9 and A-10, respectively. The HNSS has a greater resistance to stress-life fatigue than Custom 465 SS, but its resistance is similar or slightly inferior to those of the 13-8Mo and MLX17 SS in air and 3.5% NaCl solution, Figure A-9. The fatigue crack growth resistance of the HNSS is better than those of the 13-8Mo and Custom 465, but similar to that of MLX17 SS in air. On the other hand, it is similar to those of the other stainless steels, Custom 465, 13-8Mo, and MLX17 SS in 3.5% NaCl solution, Figure A-10.

CONCLUSIONS

HNSS is highly ductile and workable.

Cold-work improves the YS, UTS, and hardness, but reduces the elongation and fracture toughness. The mechanical property improvement is attributable to N-induced strengthening by interstitial solid solution, planarity of dislocation slip, deformation twins, and grain refinement.

Cold-work extends the fatigue crack initiation and total fatigue lives, but shortens the fatigue crack growth life, reducing ΔK_{th} and increasing da/dN .

HNSS, annealed or cold-worked, is immune to SCC, but susceptible to corrosion fatigue in 3.5% NaCl solution.

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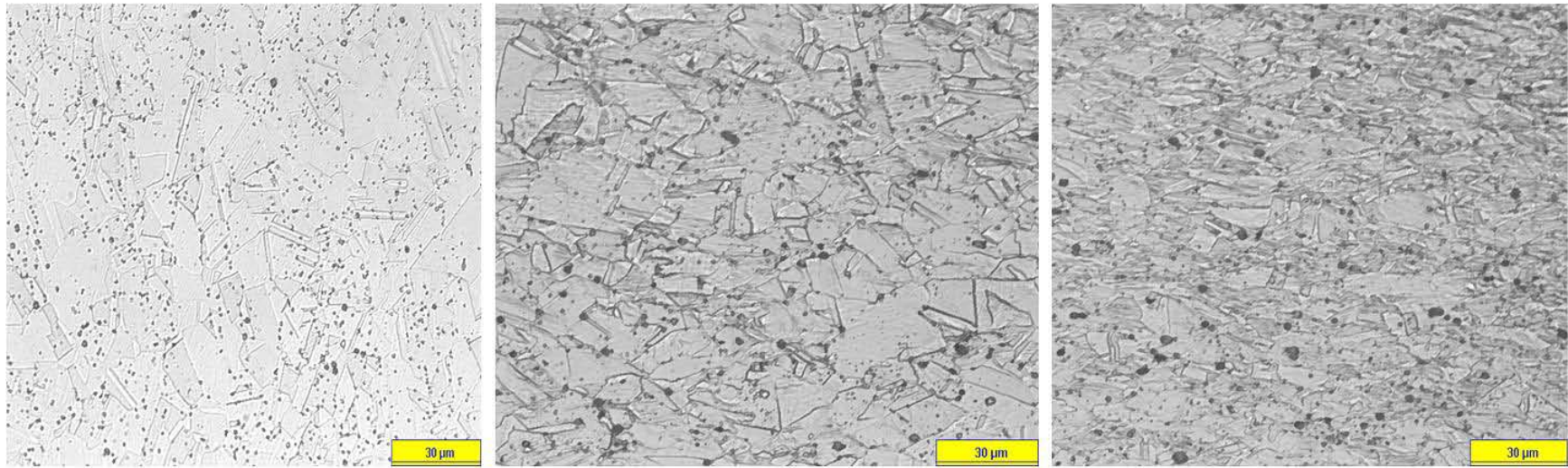
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APPENDIX A
FIGURES

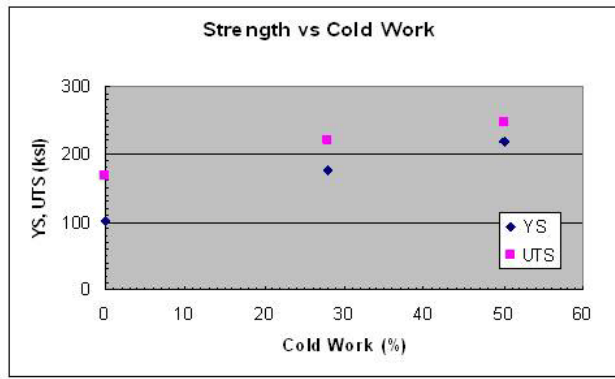


annealed

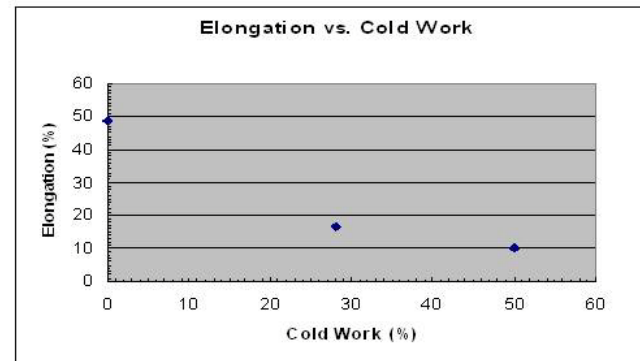
35% cold-worked

50% cold-worked

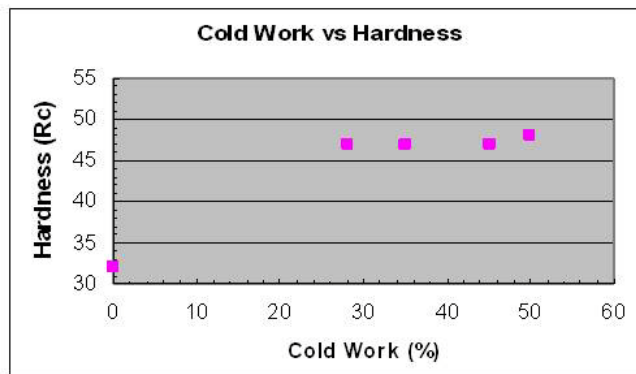
Figure A-1: Microstructures of Annealed and Cold-Worked HNSS



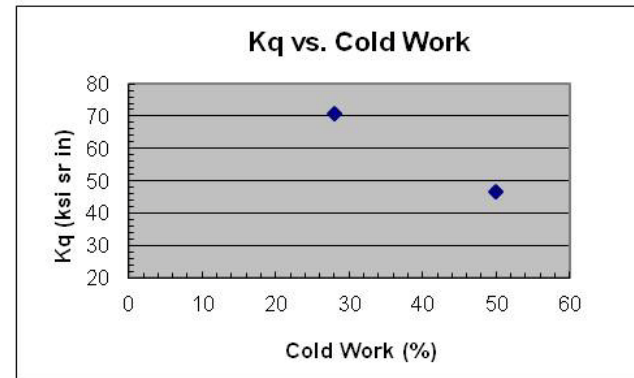
(a) YS/UTS vs. Cold-Work



(b) Elongation vs. Cold-Work



(c) Hardness vs. Cold-Work



(d) Kq vs. Cold-Work

Figure A-2: Variation of Mechanical Properties with Cold-Work

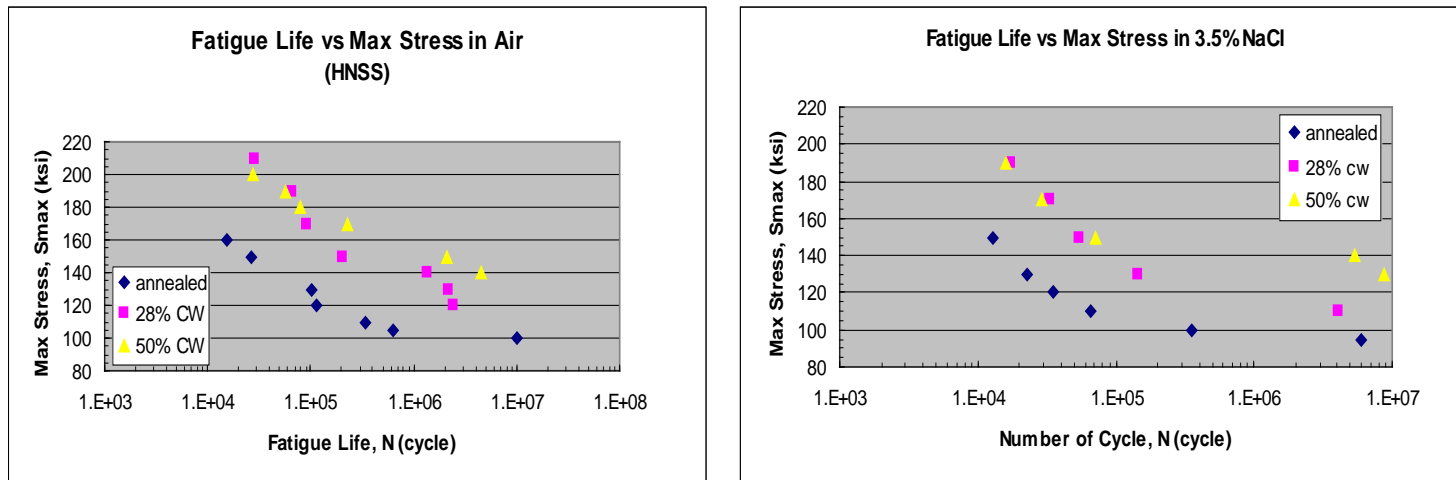


Figure A-3: Stress-Life Fatigue Curves for Annealed and 28% and 50% Cold-Worked Specimens, Tested in Air and 3.5% NaCl Solution of pH 7.3

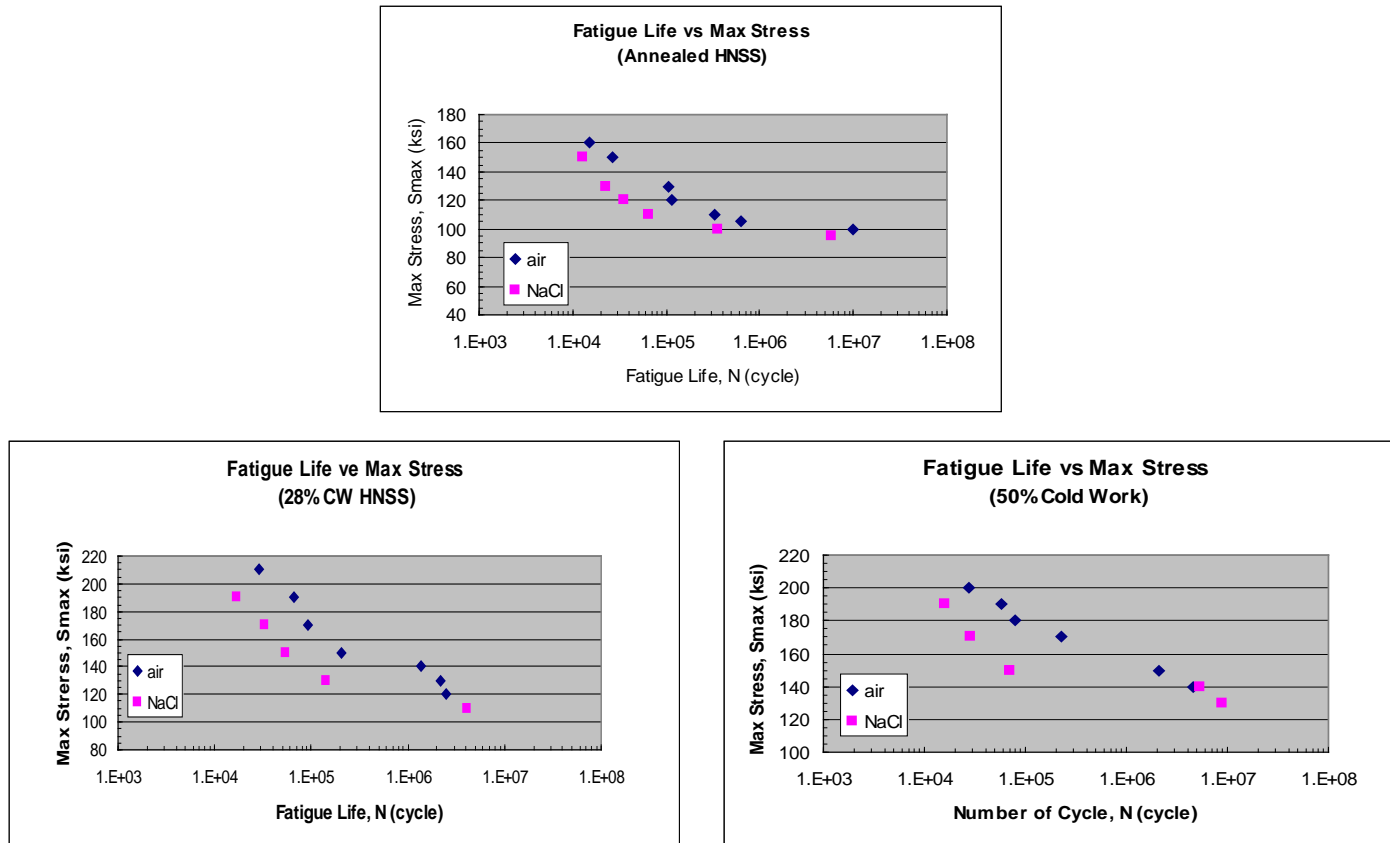
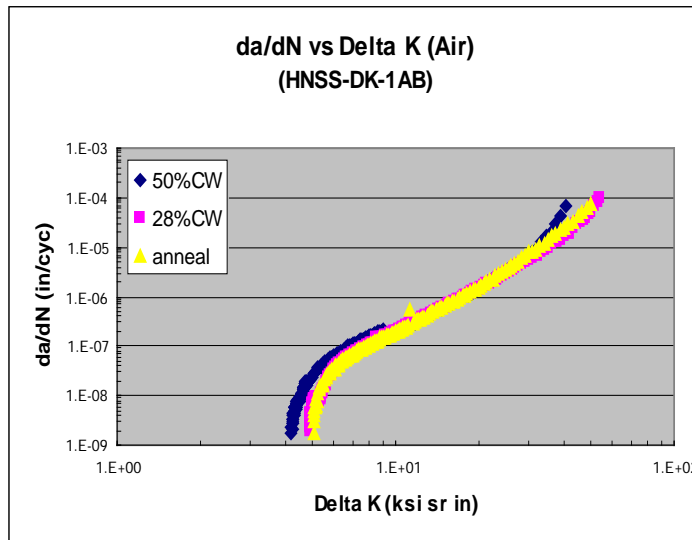
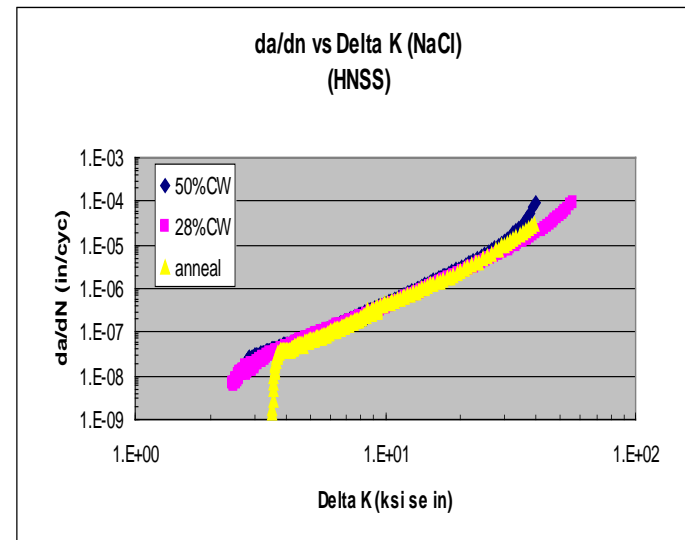


Figure A-4: Comparison of Fatigue Lives in Air and 3.5% NaCl Solution



Tested in Air



Tested in 3.5% NaCl Solution

Figure A-5: da/dN Versus Delta K for Annealed and 28% and 50% Cold-Worked Specimens,
Tested in Air and 3.5% NaCl Solution

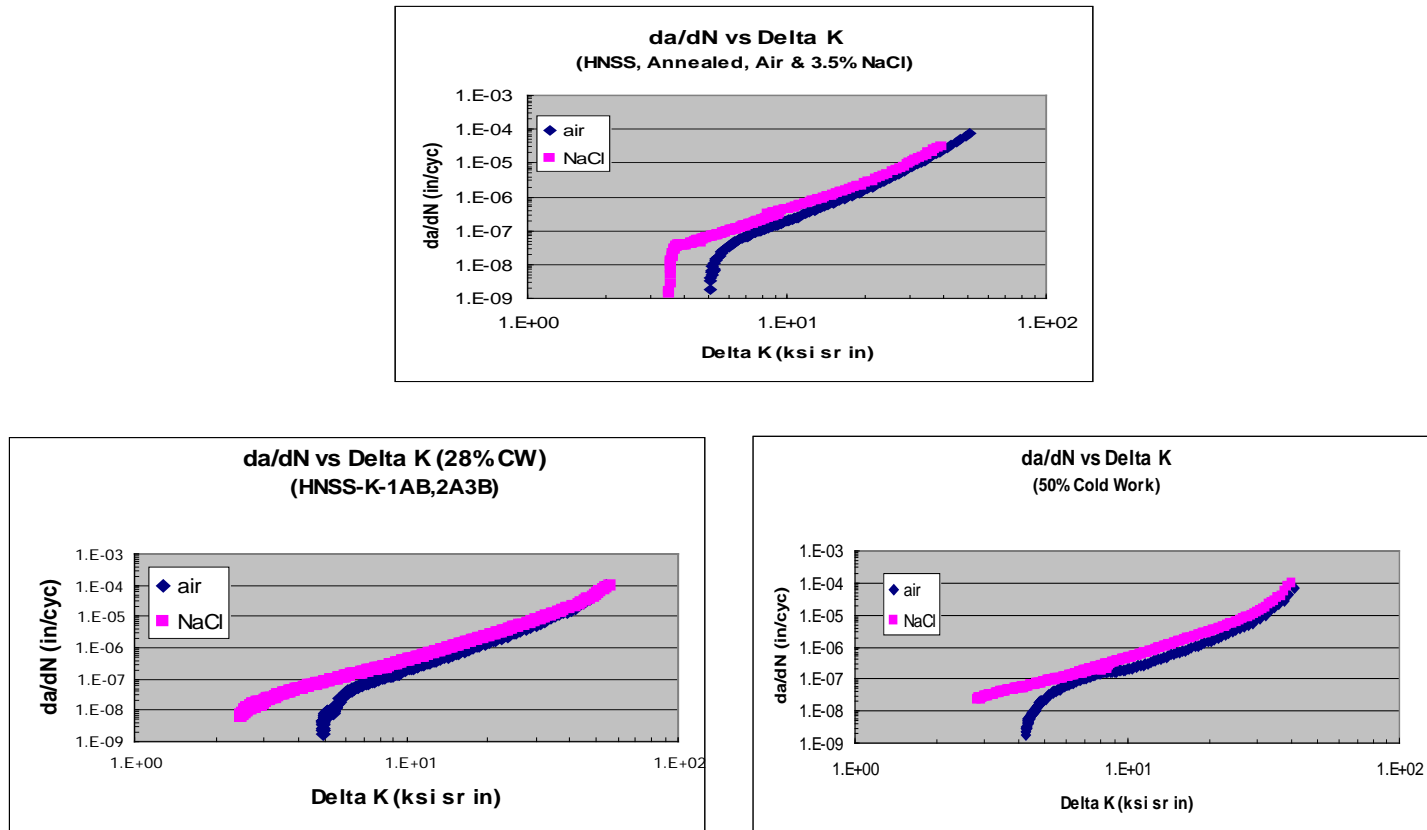
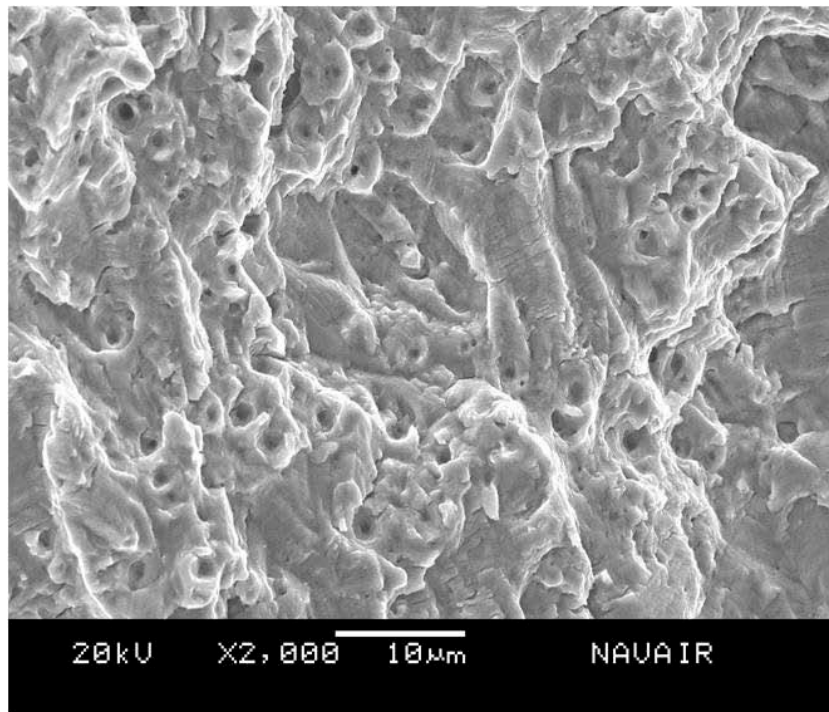
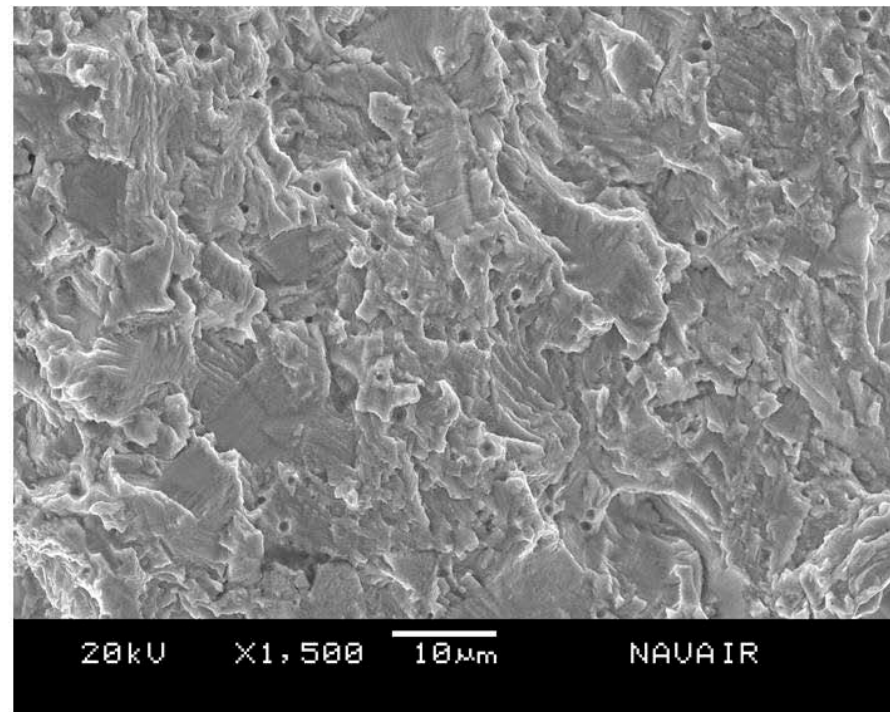


Figure A-6: da/dN Versus ΔK in Air and 3.5% NaCl Solution for Annealed and 28% and 50% Cold-Worked Specimens

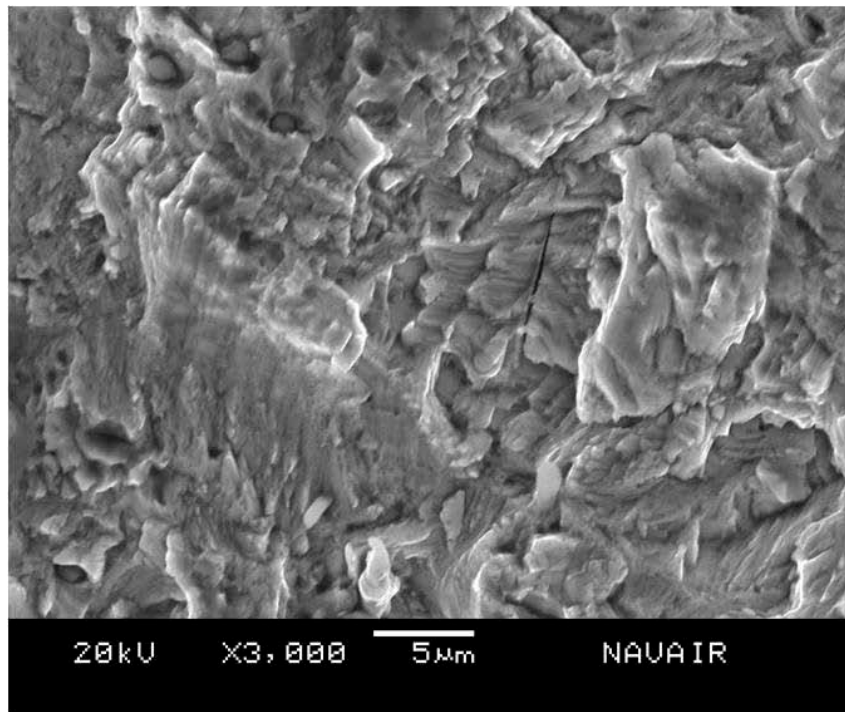


Fatigue-Tested in Air

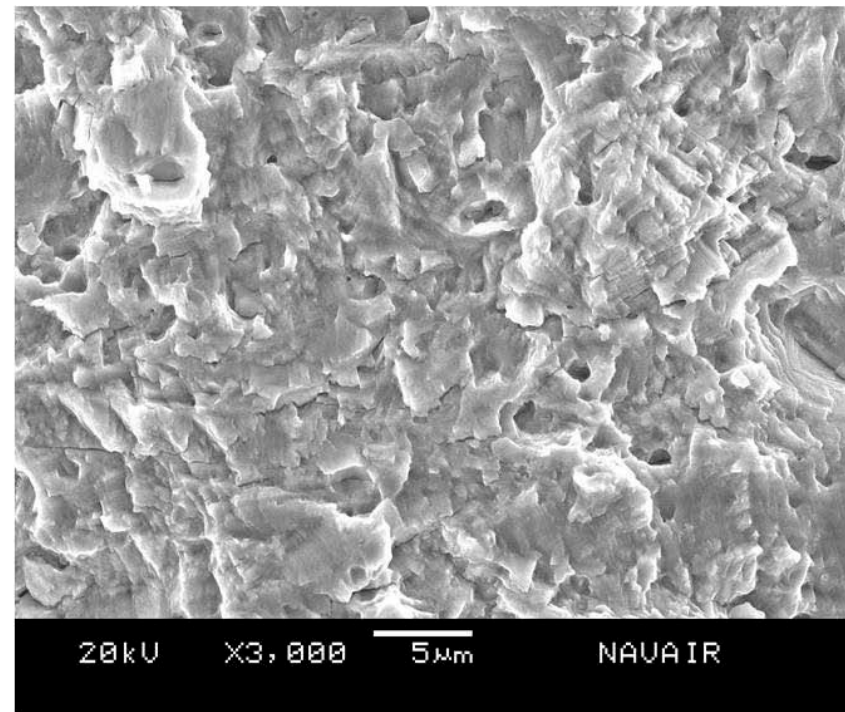


Fatigue-Tested in 3.5% NaCl Solution

Figure A-7: SEM Fractographs of Annealed HNSS Fatigue-Tested



Fatigue-Tested in Air



Fatigue-Tested in 3.5% NaCl Solution

Figure A-8: SEM Fractographs of 50% Cold-Worked HNSS Fatigue-Tested

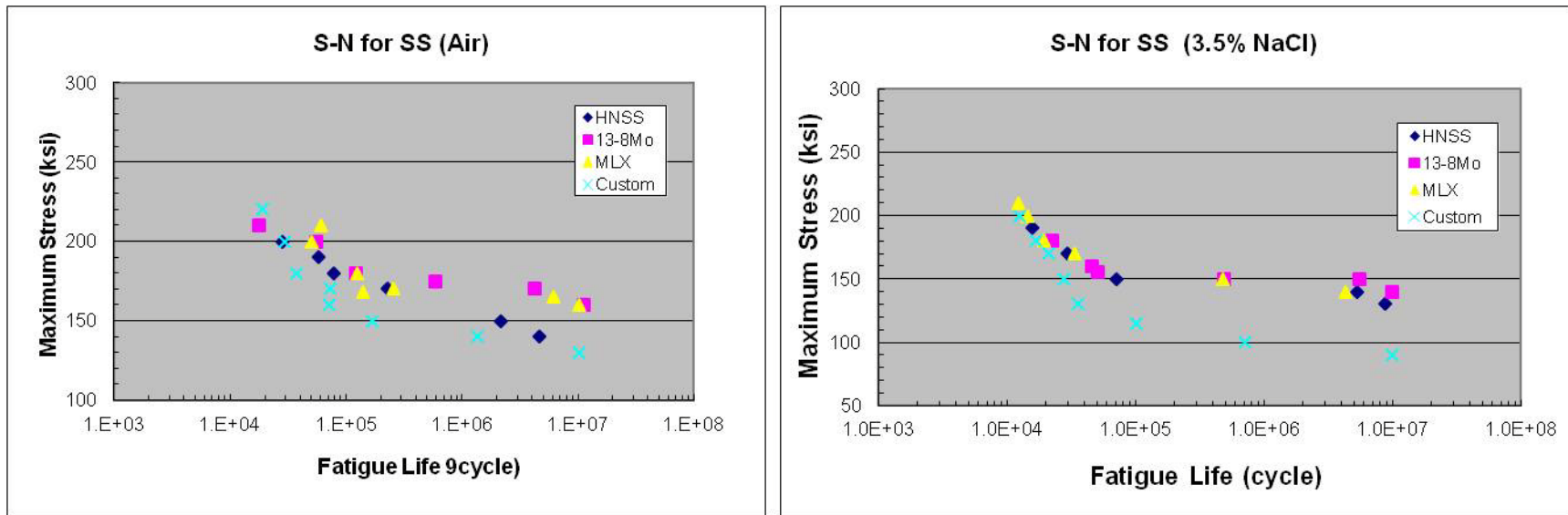


Figure A-9: Stress-Life Fatigue Curves in Air and 3.5% NaCl Solution for HNSS, 13-8Mo, MLX17, and Custom 465 Stainless Steels

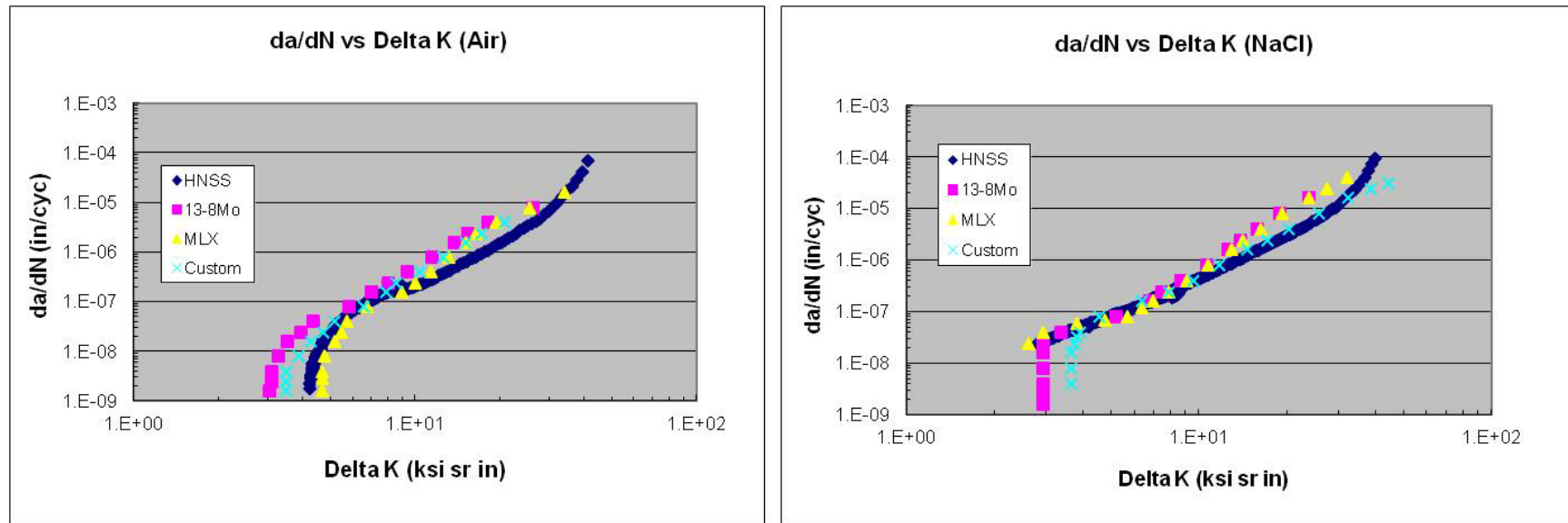


Figure A-10: da/dN Versus ΔK in Air and 3.5% NaCl Solution for HNSS, 13-8Mo, MLX17, and Custom 465 Stainless Steels

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APPENDIX B
TABLES

Table B-1: Chemical Composition of HNSS

| <u>Element</u> | <u>Composition (%)</u> |
|-----------------------|-------------------------------|
| C (Max) | 0.03 |
| P | 0.020 |
| Si | 0.50 |
| Ni | 15.00 |
| O (Max) | 0.03 |
| Mn | 6.00 |
| S (Max) | 0.004 |
| Cr | 29.5 |
| Mo | 2.00 |
| N | 0.85 |
| Fe | Balance |

Table B-2: Mechanical Properties of Annealed and Cold-Worked HNSS

| | <u>YS</u> (ksi) | <u>UTS</u> (ksi) | <u>Hardness</u> (Rc) | <u>Elongation</u> (%) | <u>Kq</u> (ksi sr in) |
|------------------------|----------------------------------|-----------------------------------|---------------------------------------|--|--|
| <u>Annealed</u> | 101 | 168 | 32 | 48 | 49* |
| <u>28% CW</u> | 178 | 220 | 47 | 17 | 71 |
| <u>50% CW</u> | 219 | 248 | 48 | 10 | 47 |

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Table B-3: Mechanical Properties of Some Stainless Steels

| <u>Property</u> | <u>HNSS</u> | | | <u>Custom 465</u> | | | <u>13-8Mo</u> | <u>MLX17</u> |
|-----------------------------------|----------------------|-----------|-----------|----------------------|------------|------------|----------------|--------------|
| | <u>Cold-Work (%)</u> | | | <u>60% Cold-Work</u> | | | <u>(H1000)</u> | |
| | <u>0</u> | <u>28</u> | <u>50</u> | <u>482</u> | <u>524</u> | <u>552</u> | | |
| <u>UTS</u> (ksi) | 168 | 220 | 248 | 307 | 282 | 263 | 215 | 248 |
| <u>YS</u> (ksi) | 101 | 178 | 219 | 304 | 279 | 258 | 207 | 243 |
| <u>Hardness</u> (R _c) | 32 | 47 | 48 | 54 | 52 | 51 | 44 | 48 |
| <u>K_Q</u> (ksi√in) | 49* | 71 | 47 | 92 | 105 | 114 | 107 | 88 |
| <u>K_{ISCC}</u> (ksi√in) | Immeasurably High | | | 21 | 54 | 84 | 77 | 75 |
| <u>Elongation</u> (%) | 48 | 17 | 10 | 12 | 12 | 14 | 14 | 14 |

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